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Techniques for Measuring Spectral Emittance of Solids at High Temperatures Using a Solar Furnace

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Three related methods were investigated to determine spectral emittances at temperatures near 2000 deg C. The work was carried out using the large solar furnace at the United States Army Natick Laboratories. A rotating chopper system was used in conjunction with a monochromator and optical pyrometer. Temperature readings were taken first using short pulses of emitted radiation and then using longer pulses of emitted plus reflected radiation. Measurements were made during exposure in the furnace of the spectral radiation (1) emitted by the sample, (2) emitted and reflected by the sample, and (3) reflected by a reference water-cooled reflector. Also, measurements of spectral radiation from an artificial black body were made. From this data the spectral emittances can be determined and the temperature determined by three calculations.

At first, large errors were experienced due to multiple reflections causing scattered radiation to enter the monochromator. The difficulties have now been largely overcome; methods have been found for correcting for the remaining scattered light.

THE determination of the normal spectral emittance of a surface $\epsilon_{S\lambda T}$ basically involves the measurements of the normal spectral radiation emitted from the sample and from a reference black body as a function of temperature.

The normal emittance is then given by

$$\epsilon_{S\lambda T} = (E_{S\lambda T})/(E_{B\lambda T}), \tag{1}$$

where $E_{S\lambda T}$ and $E_{B\lambda T}$ are the intensities of radiation per unit area emitted normal to the surface at a temperature T and in the wavelength interval λ to $\lambda + \Delta\lambda$, from the sample and from the reference black body respectively. At each wavelength two measurements of temperature and two of radiation are made.

The temperature measurement of the black body, T_B, and the emitted radiation of sample and black body can be made using well-established methods. However, at high temperatures where radiation pyrometry is required, the temperature of the specimen, T_S, is more difficult to determine. A large error can occur in the calculation of emittance for a small error in T_S. This is especially so at wavelengths shorter than the wavelengths of maximum energy. For this reason it is inherently a better technique to use a method in which the accuracy of $\epsilon_{S\lambda T}$ does not depend on the determination of temperature but rather the determination of temperature depends on $\epsilon_{S\lambda T}$.

A number of techniques using this approach and employing an image furnace have been suggested by Blau[1], Comstock[2], Laszlo, Gannon and Sheehan[3]. Methods were used to separate the emitted from the reflected radiation of the sample and measurement taken to determine reflectance and hence emittance.

Blau suggests the determination of $\epsilon_{S\lambda T}$ from measurements of

(1) The radiation emitted by sample.
(2) The sum of emitted and reflected radiations.
(3) The radiation reflected by a calibrating reflector placed in the furnace.

The determination of temperature is made by observing the apparent temperature under the above three conditions and then a calculation of true temperature T_S can be made using the emittance value above.

A practical difficulty arises in determining the apparent temperature when viewing the sample while it is

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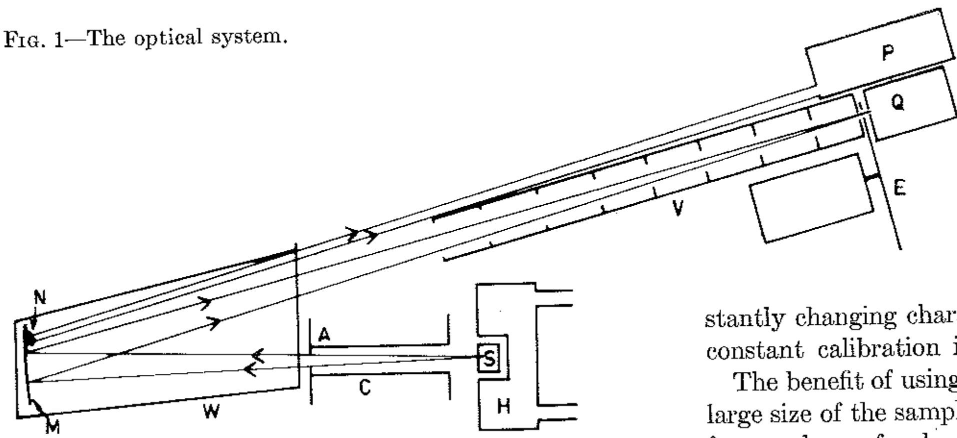
FIG. 1—The optical system.



cut off from the source. Although frequent views of the sample are possible, they are of extremely short duration. For the purpose of radiation pyrometry under these conditions, the effective transmission of the chopper system is extremely small, apparent temperatures differing from the true temperature by as much as 1000 deg C are obtained.

Comstock has used to advantage the images formed at the intermediate image plane of a double ellipsoidal carbon arc-image furnace. By making radiation-profile measurements at the intermediate image plane of the arc end of the sample, with and without incident arc radiation, he describes a method of deriving the spectral reflectance of the sample at every point across the surface. From the reflectance profile and the emitted radiation profile, the temperature profile can be deduced.

This work depends on the images at the intermediate image plane being well resolved images of the carbon arc, the specimen and the calibrating reflectance surface. Except for a very small area right at the focus, I do not think that points on the image are well resolved. When using wide-angle optics as in this case, a point not precisely at the focus is imaged at different points on the intermediate image plane by different parts of the mirror. The image formed in the plane, therefore, is a complicated composite of the object.

Laszlo, Gannon, and Sheehan have calibrated a 60-inch diameter solar furnace and successfully used this calibration instead of a reference reflection standard. It is difficult to apply this technique to large solar furnaces such as at Natick. The optical components of both heliostat and concentrator mirror are exposed to the elements. The concentrator mirror is front-surface aluminized and the reflection of each component gradually deteriorates until it is removed, realuminized and realigned. The components of the heliostat are back-surfaced mirrors so that the reflection from this surface does not appreciably change. However, the front surface collects dust and hence gradually reduces the effective reflectance until the whole mirror is cleaned. This combines to produce a furnace of constantly changing characteristics and this would make a constant calibration impossible.

The benefit of using a large furnace is obtained in the large size of the sample that can be heated. The Natick furnace has a focal area of intense irradiation of three inches in diameter. The irradiation is almost constant over a central area one inch in diameter.

Experimental Methods

The aim of this work was initially to obtain data to calculate three semi-independent sets of results for spectral emittance as a function of temperature over the spectral range 0.7 microns to 0.3 microns. With this in view, the following measurements were made:

a—The radiation emitted by the sample, $E_{S\lambda T}$.

b—$E_{S\lambda T}$ together with the solar radiation reflected by the sample, $R_{S\lambda T}$.

c—The solar radiation reflected, $R_{C\lambda T}$, by a water-cooled calibrating reflector.

d—The apparent temperature, T_1, of the sample using emitted radiation only.

e—The apparent temperature, T_2, of the sample, determined using emitted plus reflected radiation.

f—The emitted radiation, $E_{B\lambda T}$, by a black body.

The emittance from the sample determined from (a), (b), and (c) is given by:

$$\epsilon_{S\lambda T} = 1 - \frac{[(E_{S\lambda T} + R_{S\lambda T}) - E_{S\lambda T}]}{R_{C\lambda T}} r_{C\lambda T} \qquad (2)$$

where $r_{C\lambda T}$ is the reflectance of the calibrating reflecting surface.

The true temperature can be determined by three methods as follows:

a—Correct T_1 due to the transmission of the chopper system and due to $\epsilon_{S\lambda T}$ for the working wavelength of the pyrometer.

b—Calculate an effective emittance for the correction of T_2. The radiation received by the pyrometer is proportional to $E_{S\lambda T} + R_{S\lambda T}$. However, it has been determined that $E_{S\lambda T}$ represents an emittance of $\epsilon_{S\lambda T}$. Therefore, radiation coming from the sample of $E_{S\lambda T} + R_{S\lambda T}$ for optical pyrometer correction purposes represents an emittance of ϵ_2, where

$$\epsilon_2 = \epsilon_{S\lambda T} \frac{E_{S\lambda T} + R_{S\lambda T}}{E_{S\lambda T}} \qquad (3)$$

c—The temperature is calculated independent of

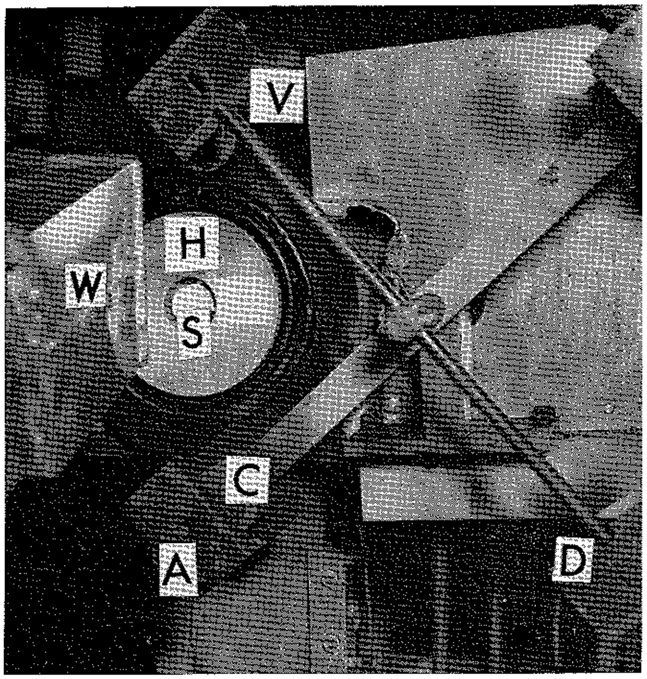


FIG. 2—Part of the optical system with target in position.

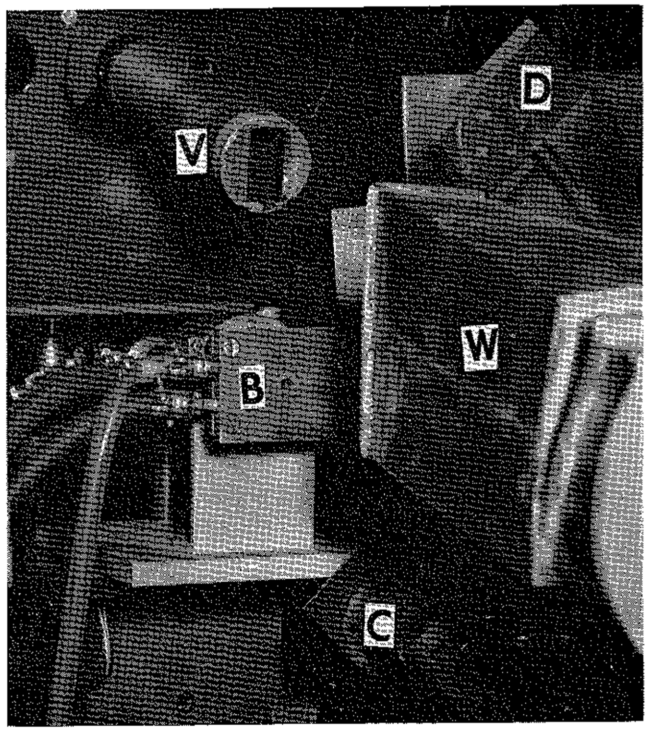


FIG. 3—A second view of the optical system with the black body in position.

pyrometer readings. The energy that would be emitted by a black body at the same temperature as the sample is given by $(E_{S\lambda T})/(\epsilon_{S\lambda T})$. By now referring to the black-body curves of energy vs temperature for the wavelength being used, the true temperature can be obtained.

Equipment and Procedure

The experimental equipment is illustrated in Figs. 1, 2 and 3. The sample, S, is supported in a polished aluminum cavity by alumina or zirconia rods that pass through sleeves in the water-cooled holder, H. The sample is a rod one inch in diameter and $\frac{3}{4}$ inch long. The polished cavity walls partially surrounding the sample help reduce temperature gradients by reflecting solar radiation to the side and back surfaces of the sample as well as reflecting some of the emitted radiation back to the sample.

For use as the reference reflectance standard, an area about 1.5 inches diameter of the face of the water-cooled sample holder is smoked by burning magnesium ribbon.

The holder is mounted on a platform so that it can be moved quickly from the position where the sample is under observation to the position where the magnesia surface is observed. To guard against errors due to a change in the intensity of solar irradiation, this cycle can be repeated as required.

The Chopper System

The primary chopper has four arms, two of which, C, are designed to cut off the incident light and allow the passage of the emitted beam through an aperture, A, to a concave mirror, M, which is inside the water cooled shield, W. After reflection by M the beam passes through a pipe, V, and synchronized chopper, E, to form an image of S on the entrance slit of a monochromator, Q.

The other two arms, D, of the primary chopper do not interrupt the incident beam and through an aperture in them the monochromator receives emitted plus reflected radiation.

The chopper, E, is essentially a disc with four holes which synchronize with the aperture in the four primary chopper arms.

The primary chopper is driven by a variable-speed, direct-current motor and E is electrically coupled to the motion of the primary. To prevent the disc from hunting, it was necessary to increase its moment of inertia and to increase its resistance to motion by attaching a small fan.

The chopping system is usually set rotating at 400 rpm and the corresponding signal length is 3 milliseconds.

The emitted radiation is much less than the emitted plus reflected radiation and as they are recorded alternatively in rapid succession, it was found expedient to have different sized apertures in C and D. In order to have the recorded signals of approximately the same height, two large apertures are available for C and seven smaller apertures for D from which a selection of the most suitable two can be made.

The Monochromator and Recorder

The monochromator is a Ferrand UVIS, Catalog No. 103420, with a 14,400 lines per inch reflection-type replica grating. It has two ranges, one from 0.25 microns to 0.7 microns, using 1P22 photomultiplier sensor and another from 0.7 microns to 2.7 microns using a lead-sulphide sensor. The output signals are amplified by a Consolidated Electrodynamics Corporation (CEC) type 1-155 multirange amplifier, and recorded by a CEC Model 5-124 recording oscillograph. The galvanometer used in the recorder has a resonance frequency of 5000 cycles per second and ample sensitivity. Using a chart speed of 64 inches per second, the millisecond signals are well displayed.

The Black Body

The black body consists of an electrically heated graphite tube mounted inside a water-cooled cylindrical jacket. The inside of the jacket is polished to reduce heat losses from the tube and to assist toward a uniform temperature throughout it. Electrical terminals are attached to each end of the tube and alternating currents up to 1300 amperes at 10 volts are used producing a temperature to 3400 deg C. The variable-output power supply consists of a variac and transformer connected to a 440-volt outlet.

The graphite tube is of length 2.25 inches, outside diameter 0.50 inches to 0.56 inches, and inside diameter 0.28 inches. A 0.25 inch diameter septum was pushed midway down the tube and measurements were made viewing the septum through one end of the tube. It was noticed that due to cooling at the two ends there was a temperature gradient along the tube and black body conditions were not obtained. This was partially rectified by tapering the walls of the tube so that they were thicker near the middle, thus producing a higher electrical resistance and therefore more heat energy near the ends and so reducing the temperature gradient. Further, a slot was cut in the septum $\frac{1}{16}$ inch wide and $\frac{3}{16}$ inch deep. However, when using a new tube a temperature gradient still exists with the septum at a higher temperature than the end walls of the tube. The slot in the septum is seen as a bright line due to its greater emittance compared with the remaining surface of the septum. In use the sleeve oxidizes more rapidly near the exposed end and the temperature of this end slowly increases above that of the septum. The slot now appears as a dark line due to its relatively low reflectance.

For temperature and emission readings on the black body, it is placed in the sample position, B, Fig. 3, and the same optical and recording system used as for the sample.

Temperature Measurement

The measurement of T_1 required another chopper in front of an optical pyrometer, P, in phase with C so that the pyrometer received short pulses of emitted light only. This method was considered rather inaccurate due to the large corrections required and so was not continued.

The measurement of T_2 is made without the use of a chopper in front of the pyrometer. By means of a small mirror, N, the pyrometer views the sample or the black body except when the rotating primary chopper intercepts the light.

Using the black body and alternately viewing it through the rotating chopper and then with an uninterrupted view, it was found that for pyrometer readings an emittance correction of 0.40 was necessary to correlate the two sets of readings. This means that when viewing the sample, the apparent emittance ϵ_{app} is given by:

$$\begin{aligned}\epsilon_{app} &= 0.40\epsilon_2 \\ &= 0.40\epsilon_{S\lambda T}\frac{E_{S\lambda T} + R_{S\lambda T}}{E_{S\lambda T}}\end{aligned} \tag{4}$$

Also, when viewing the black body through the rotating chopper, a correction must be made for an apparent emittance of 0.40.

Shielding

In the Natick Laboratories furnace, up to 30 kw of radiant energy enters the working chamber. Some of this energy, after one or more reflections, is likely to enter the monochromator and early results were meaningless due to this. Rather elaborate precautions are necessary to reduce this effect to a low level.

The water-cooled shield, W, was placed around the mirrors, M and N, with an aperture only large enough to transmit the necessary radiation. The pipe, V, with a blackened and baffled interior, prevents some of the stray radiation reaching the monochromator. The choppers are blackened first by a matte-black paint and then coated with carbon black. Further, some surfaces of the choppers were covered with an aluminum honeycomb before blackening. This produced a surface reflectance of probably less than one percent.

There still remains a small error in the signal due to spurious reflections. This is measured continually by blocking the apertures in one of the choppers, C, and in one of the choppers, D. It is assumed that the error due to spurious reflections can be eliminated by the subtracting of signals obtained with the blocked choppers from the corresponding signals with the open choppers.

Actual traces when viewing the specimen and the magnesium oxide surface respectively are shown in Figs. 4 and 5. In Fig. 4 the corrected emission signal is given by the difference between the two C signals; the corrected emission plus reflection signal is given by the difference between the two D signals. In Fig. 5 the corrected magnesia signal is the difference between the two D signals.

Aperture Calibration

Using the black body as a source, curves were drawn of emitted energy vs temperature at fixed wavelength intervals for each of the different chopper apertures. These were then compared with standard black-body curves and a corrected figure obtained for the emitted energy.

By using the smaller aperture the measured output would fit the theoretical curves very accurately. However, with the larger apertures there was some departure from the theoretical curves at higher temperatures. This is thought to be due to partial saturation and subsequent non-linear output of the photomultiplier tube and associated circuit.

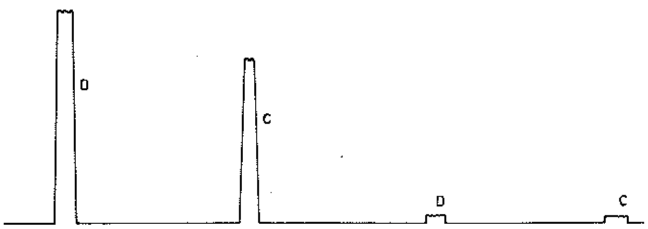


FIG. 4—A tracing of an oscillogram obtained with the sample in position showing the emitted signal C and the emitted plus reflected signal D with their respective corrections.

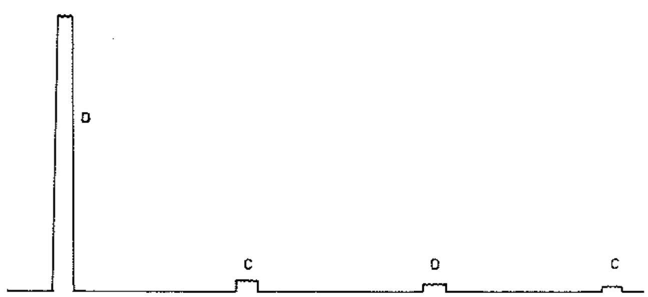


FIG. 5—A tracing of an oscillogram obtained with the calibrating surface in position showing the reflected signal D with its correction.

Sample—The samples used are cylindrically shaped pieces of coarse granula 90 percent zirconia. Using a diamond cutting wheel, $\frac{3}{4}$-inch lengths are cut from a one-inch diameter rod. The flat ends are used for the radiation and temperature measurements without further treatment.

Results—Data have been taken over the wavelength range 0.25 microns to 0.665 microns at intervals of approximately 0.050 microns, and over a temperature range from 1950 to 2200 deg K. Only the initial sets of data at 2100 deg K approximately in the range 0.450 microns to 0.665 have been used to compute actual emittances and temperatures. The mean values obtained are:

Wavelength, microns	Emittance ± 0.05
0.665	0.77
0.600	0.82
0.550	0.81
0.500	0.84
0.450	0.86

The variation in emittance results leading to an estimated error of 5 percent was at least partially caused by the coarse texture of the samples used. The area observed is 0.125 inches long and 0.020 inches wide and the same area was not observed when obtaining each set of data.

The temperature calculated from the black-body emission curves and those determined by an emittance correction, or T_2, agreed very well. The mean values of the two sets of temperatures differed only by 5 deg C. Individual pairs of readings differed, however, by as much as 15 deg C.

Some errors due to spurious reflections are not measured by the blocked choppers. One source of such an error can be caused by reflections in turn from the target holder to the near flange of chopper C, to the sample and finally from the sample through the chopper with the emitted radiation. To reduce this possibility, the chopper flange was made larger and a blackened honeycomb mesh attached to it.

These remarks indicate some of the problems involved in this application of a solar furnace. While some problems remain, there appears to be no unsurmountable difficulty to obtaining accurate spectral emittance data up to the maximum temperatures attainable by the furnace.

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